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A systematic study of the residence time of flour in a vibrating apparatus used for thermal processing

S. Keppler [a,*], S. Bakalis [a], C.E. Leadley [b], P.J. Fryer [a]

[a] *University of Birmingham, School of Chemical Engineering, Edgbaston, Birmingham, B15 2TT, UK*
[b] *Campden BRI Gloucestershire, UK*

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ABSTRACT

The dry heat treatment of flour is well established for the production of cake flour for high ratio cakes. This study investigates a new tubular apparatus in which flour is conveyed by vibrations through a helical pipe. Residence time distributions (RTDs) of flour were characterised for various processing conditions and the development of the residence time in extended operation was analysed.

A method was developed to accurately determine the RTDs, which could be approximated by normal distributions. The width of the distributions is a critical factor for the accuracy of a thermal process and was identified for different processing conditions. The distributions were narrow, with variations of $\pm 1\%$ at most.

In some cases, the residence time increased over 3.5 h of machine run-time by 7.7%–13.9%. To explain this phenomenon, several hypotheses have been tested. The machine performance was constant with time and no influence of ambient temperature or humidity could be found. It was furthermore shown that changes in the bulk material passing through the apparatus were not the cause of the increase. However, electrostatic charging of the material was observed.

Two things led to a reduction in residence time: i) cleaning the pipe with a cleaning pig and water and ii) time, during which the machine is not running. It was suggested that a thin layer of particles inside the pipe in combination with electrostatics effects could be the reason for the residence time increase. Frequent cleaning can therefore allow relatively uniform behaviour and control of residence time.

Industrial relevance: This work investigates the potential application of a novel, vibrating device for the dry heat treatment of flour as a replacement for chlorination in the production of cake flour. Since chlorination was banned in the EU in the year 2000, there is an industrial interest for alternative treatments and equipment to produce flour for high ratio cakes.

1. Introduction

High ratio cake formulations are widespread in the UK in the production of sponges (e.g. Madeira cake), Angel cake, gateaux, slab cakes, or cupcakes (Hodge, 1975). Their sweet and moist characteristics achieved by a sugar to flour ratio of 1.0–1.4 are well appreciated by the market (Chesterton, Wilson, Sadd, & Moggridge, 2015; Guy & Pithawala, 1981; Magee & Neill, 2011). In comparison, cakes with lower or equal amounts of sugar and flour refer to low ratio cakes (Wilderjans, Luyts, Brijs, & Delcour, 2013). To achieve the desired attributes in high ratio cakes, it is required to use flour with a distinctive functionality to carry more water and sugar compared to flour used in low ratio cakes (Collyer, 1968). This was formerly accomplished by treating flour with chlorine gas (Collyer, 1968; Hodge, 1975).

During the baking process of a high ratio cake, the viscosity of the batter increases due to starch gelatinisation and protein coagulation (Cook, 2002). The key element is that previously introduced bubbles by water vapour, heat expansion, and CO_2 expand and eventually burst due to increasing internal pressure transforming the viscous foam structure into a solid foam with uniform air cells distributed evenly throughout the cake (Cook, 2002; Meza et al., 2011). Untreated flour does not provide the necessary batter viscosity for the bubbles to rupture and the cake collapses when the temperature falls on removal from the oven (Cook, 2002). In contrast, chlorination increases the swelling capacity of the starch granules and the hydration capacity of gluten ensuring the mutual contact between starch granules after gelatinisation, increasing batter viscosity, allowing the cake structure to solidify in the final stages of baking and thus preventing the cake from collapsing (Collyer, 1968; Gough, Greenwood, & Whitehouse, 1977; Guy & Pithawala, 1981).

Whereas chlorination of flour is still used in the US, the process was banned in the EU in 2000 after concerns about health risks were raised

* Corresponding author. Tel.: +44 7557651407.
E-mail address: sxk250@bham.ac.uk (S. Keppler).

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(Catterall, 2000). Two general alternatives appeared to be other treatment processes or the modification of cake formulations (Gough et al., 1977).

In this context, Mangels in 1934 had discovered that the dry heat treatment of starch increased its rate of swelling in dilute sodium hydroxide solution (Hodge, 1975). This is the basis of the current heat treatment process of flour in industry, which generally involves 3 thermal steps followed by rehydration and milling (Chesterton et al., 2015). Specific details of flour heat treatments are not readily available in the public domain. In a first drying step, the flour is heated up and the moisture content is reduced, preferably below 4% in a stream of hot air (Chesterton et al., 2015; Neill, Al-Muhtaseb, & Magee, 2012). In a second heat treatment step, the flour is held at high temperatures by contact heating in rotated drums or heated conveyors (e.g. 120 °C–140 °C, 20–30 min) (Chesterton et al., 2015; Doe & Russo, 1970). After a cooling step to interrupt the heat treatment, the flour is rehydrated to a moisture content of e.g. 7%–12% (Chesterton et al., 2015; Neill et al., 2012). A final milling step is generally applied to break up agglomerates formed during rehydration (Chesterton et al., 2015).

As the dry heat treatment of flour is a physical modification, it is broadly accepted in public in contrast to chemical methods (Hodge, 1975; Thomasson, Miller, & Hoseney, 1995). Little is published about the alterations in flour generated by heat treatment, but the key effect of flour improvement appears to relate to the surface of the starch granules and its neighbouring layers (Guy & Pithawala, 1981; Magee & Neill, 2011). Two major effects of flour improvement by heat treatment were specified (Cook, 2002; Guy & Pithawala, 1981):

i) Improved swelling power of the starch granules and thereby increasing the batter viscosity
ii) Increased interaction between the starch and egg proteins, increasing the gel firmness for batters of treated flours.

In this study, a novel machine (Revtech, 2015) is presented that can potentially be used for the continuous heat treatment of flour. The core piece consists of a helical tube that is heated via resistive heating and that conveys the product from the bottom to the top of the spiral by vibrations. With the aim of designing a uniform and efficient process it is essential to investigate residence time distributions of the product passing through the machine as well as to examine the applied temperature profiles. The present work focuses on the residence time distributions depending on various processing conditions and their peculiarities with respect to the dynamics of the system.

2. Materials and methods

2.1. Material

Commercially available high ratio flour (protein content 8.6%) with the particle size distribution shown in Fig. 1 (calculated from triplicates) was used for the experiments. Bulk density and tap density were measured to be 0.51 ± 0.003 g/ml and 0.81 ± 0.01 g/ml.

2.2. Equipment

The equipment used for the experiments is a continuous, thermal processing unit manufactured by Revtech process systems (Loriol-sur-Drome, France). It consists of three major parts, a hopper, a heating spiral, and a cooling spiral. In this study only the hopper and the heating spiral were used for simplicity. As shown in Fig. 2, particles are conveyed by a screw feeder (B) from the hopper (A) into the spiral. It is a helical, steel pipe (C) with an internal diameter of 84.5 mm, a slope of 2.83° to the horizontal, and a length of approximately 34.4 m. It is possible both to heat the pipe by resistive heating and to inject steam. The screw speed controls the overall particle flow, whilst the behaviour in the tubing is controlled by the vibrations of the motors. Two off-balance motors (D) are attached on opposite sides of the spiral and at an adjustable angle with the horizontal. They create vibrations of different amplitudes and frequencies. The vibrations can be controlled by changing:

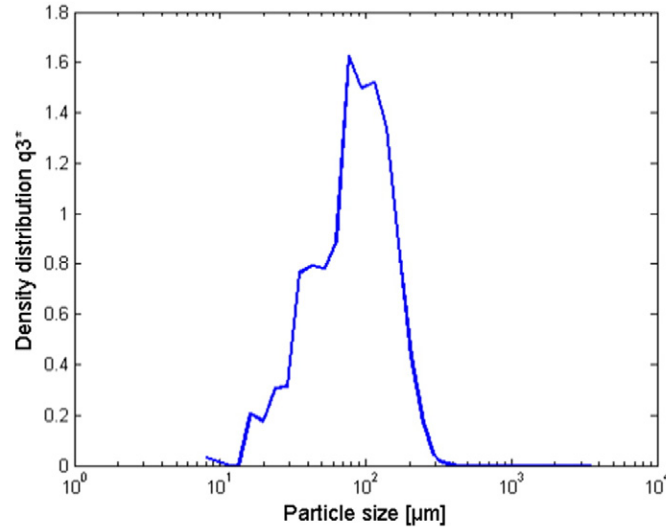

Fig. 1. Particle size distribution of high ratio flour.

- *Motor angle*. The movement of the motor is perpendicular to the motor axis. Adjustment of the motor angle β affects the velocity components of the motors in horizontal and vertical direction. This is shown in Fig. 3 and the components are defined as Eqs. (1) and (2). With increasing angle β, which ranges between 0° and 90° ($\alpha = 90° - \beta$), the horizontal velocity component increases, whereas the vertical one decreases.

$$v_x = v \cdot cos(\alpha) \quad (1)$$

$$v_y = v \cdot sin(\alpha) \quad (2)$$

- *Motor speed*. This controls the vibrational frequency of the oscillations and can be adjusted between 600 rpm (10 Hz) and 740 rpm (12.3 Hz).

The vibrations cause particles to move from the bottom to the top of the spiral. The product leaves the helical pipe via a flexible plastic tube that is connected to the top and it is collected in a plastic container.

For the experiments, the hopper of the machine was typically filled with approximately 100 kg of flour, which allowed for a 1 h experiment (100 kg/h). The flour was recirculated and the fraction lost (ca. 5%) through sample taking was topped up prior the next experiment (recirculation method).

A dry and a wet cleaning method can be applied to the spiral. A cleaning pig is used in both cases to push the remaining product out of the pipe. Compressed air is used in one case and water in the other to drive the cleaning pig through the pipe. In one case the hand dishwashing liquid Suma Star plus D1 plus (JohnsonDiversey, Inc.) was added to clean the pipe.

2.3. Determination of residence times

2.3.1. Residence time distributions

The residence time distributions of flour in the vibrating machine were measured at ambient temperature. Twenty-five grams of burnt flour (15 min at 250 °C in a convection oven) was used as a marker that was introduced instantaneously to a constant flour flow of 100 kg/h (insertion point E in Fig. 2). The time was taken with a

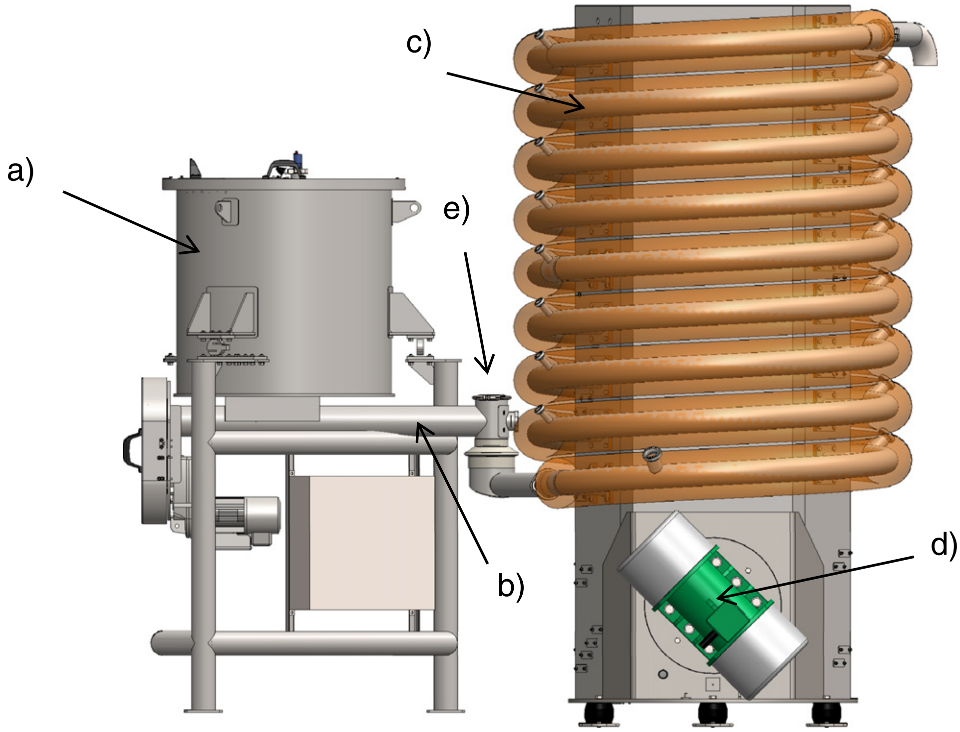

Fig. 2. Experimental setup: hopper (a), screw feeder (b), insulated helical pipe (c), motor at an angle with the horizontal (d), and insertion point for marker sample (e).

stopwatch and the marker was collected at the outlet of the flexible tube in 1 s bins. This was accomplished by moving the unit with the plastic dishes (cf. Fig. 4) by one plastic dish per second manually. The approximate residence time at which the marker was to be expected was determined in preliminary experiments.

The mass as well as the colour values *X*, *Y*, and *Z* (Konica Minolta spectrophotometer CM-5, reflectance mode, observer angle 10°, illuminant D65) of all collected bins were identified. The concentration (wt.%) of marker in each flour sample was calculated by means of a calibration curve. By multiplication of sample mass and marker concentration, the marker mass per bin was generated and related to the total marker mass collected at the outlet. Subsequently, residence time distributions were created in Minitab 17®.

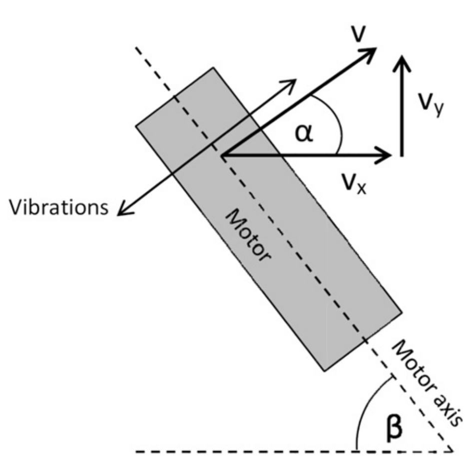

Fig. 3. Relation between total velocity (v) of the motor movement, velocity components in horizontal and vertical direction (v_x, v_y), and motor angle to the horizontal (β).

2.3.1.1. Calibration curve. To create the calibration curve, the *X*- and *Z*-values of known concentrations (wt.%) of burnt flour within a burnt flour-fresh flour-mixture were studied. Two parameters were used instead of one to increase the accuracy. *X*- and *Z*-values of the known concentrations (0–30%) were plotted in a 2D graph and a linear regression was performed (Fig. 5a). The distance α along the regression curve was calculated for the know concentrations. As the data points do not lie perfectly on the calibration line, the nearest point was found by dropping a perpendicular onto the line. α is calculated by the dot product of the unit vector of the regression curve and the vector of the measured sample (Eq. (3) m:gradient of regression line).

$$alpha = \frac{1}{\sqrt{1+m^2}} \begin{pmatrix} 1 \\ m \end{pmatrix} \circ \begin{pmatrix} X \\ Z \end{pmatrix} \tag{3}$$

α is then plotted against the corresponding concentrations of burnt flour-mixtures. A third order polynomial can be fitted to the data (Fig. 5b).

For the residence time experiments, *X* and *Z*-values were found experimentally, α calculated and the marker concentration found from the calibration curve.

2.4. Residence time dynamics

In the study of the development of the residence time with time, the residence time is taken to be the point when the marker starts to

Fig. 4. Flour collecting device for the determination of residence time distributions.

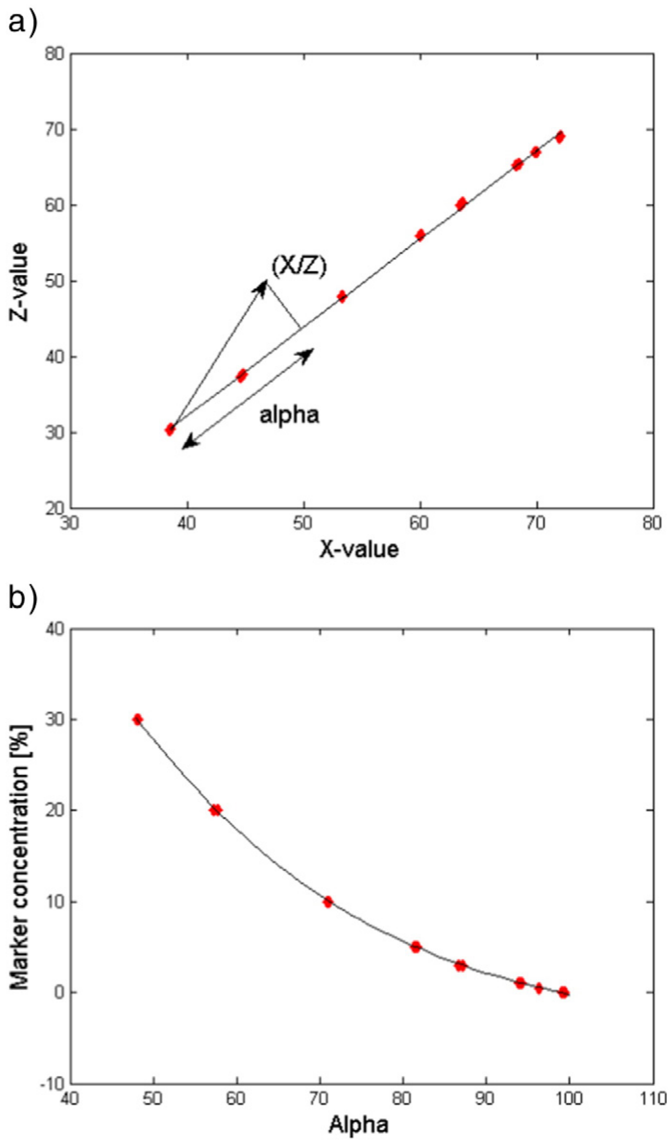

Fig. 5. Calibration curves for burnt flour-fresh flour-mixtures of concentration of 0%, 0.5%, 1%, 3%, 5%, 10%, 20%, 30%. a) *Z*-values against *X*-values. b) Concentration (wt.%) against alpha.

emerge at the outlet of the spiral. This avoids the need to measure full RTDs; Fig. 8 shows that the distributions are very narrow at all processing conditions, so the approach is justified. Thus, the first sample bin that contains marker particles is determined by eye and the corresponding time is referred to as residence time. Hence, this represents a measure of the shortest residence time. Generally, marker samples were introduced every 10 min, which allowed for 5 samples per experiment (100 kg/h). Three to 4 experiments were performed per day.

2.5. Particle size measurements and fractionation

Flour samples were taken at the outlet of the flexible tube. To avoid segregation in the sample bags, the samples were poured onto neat heaps and divided in 4 equal quarters. Three of these quarters were used individually to measure the particle size distributions on a QICPIC (Sympatec GmbH, D) with a measurable particle size of 10 μm–3410 μm (Oasis Rodos disperser, M7 lense). The 10%, 50%, and 90% quantiles of the sample volume were calculated.

Furthermore, 60 kg of flour was sifted on a Sievmaster (Farleygreene Ltd., UK) on a sieve with a hole size of 75 μm and a diameter of approximately 52 cm. Approximately 4 kg was sifted at once for 2 h. Subsequently, the residence times of the resultant fine and coarse fraction were measured.

2.6. Determination of water content

The water content of flour samples was determined in a convection oven at 130 °C according to AACC International Method 44–15.02 (note: silica gel was used as desiccant).

2.7. Charge measurements

A flour sample was collected at the outlet of the flexible pipe with a commercially available Faraday pail JCI 150 (Chilworth Technology Ltd., UK). A self-built Faraday pail was used to take samples at the inlet of the spiral due to the narrow dimensions between screw feeder and spiral inlet. Typically, a Faraday pail consists of a metal cup located within a metal shield. Both are separated by an insulator like PTFE or Teflon. The core of a coax cable is connected to the pail and the outer conducting sheath to the shield. The charge measuring unit JCI 178 (Chilworth Technology Ltd., UK) was connected to either of the pails via a bnc connector. In preliminary experiments, it was assured that both pails read comparable values. The flour mass in the cup was measured and put in relation to the charge.

The unit was zeroed briefly before the measurements and movements of the cable were avoided as this affects the charge reading. To avoid further disturbances during the measurements, full cotton clothes were worn by the person performing the experiments and additionally, the person was earthed by a wristband that is connected to earth with a crocodile clip.

2.8. Statistics

Minitab 17® was used for standard statistics, ANOVA, correlation tests, histograms and *Q*–*Q* plots. Regressions were performed in Matlab®.

3. Residence time results and preliminary discussion

For all experiments, the particles travel through the vibrating system at a flow rate of 100 kg/h in a homogenous layer with a low bed depth. The flow does not take up the entire space of the pipe, but the particles only bounce a few millimetres high.

Note that the experiments were performed on a pilot plant device and the results do not directly apply to industrial machines.

3.1. Residence time distributions

This section investigates the residence time distributions of flour at motor angles of 20°, 30°, and 40° at a motor speed of 740 rpm. Fig. 6 shows examples of collected data and fitted normal distributions. By means of *Q*–*Q* plots it was established that in all cases deviations from Normality are small (cf. Fig. 6). For 30° and 40°, the Normal overestimates the proportion of short times in the end of the distribution and the measured data shows greater residence times than predicted.

Typical normal distributions are shown in Fig. 7 standardised by dividing by the mean residence time. For 20° and 30°, the standard deviation is between 0.45% and 0.70%, whereas for 40° it is 0.70%–0.99%. Thus, the residence time distributions are narrower at lower motor angles with respect to the mean. However, the absolute spread in seconds is smaller at 40° because the residence time is shorter.

The overall mean for the data of Fig. 7 was calculated to be 548.9 s, 311.2 s, and 218.5 s for 20°, 30°, and 40°. The residence time decreases by approximately 43% (238 s) from 20° to 30° and by approximately 30% (93 s) from 30° to 40°. As the residence time was observed to shift over time as analysed below, this data only serves as an indication

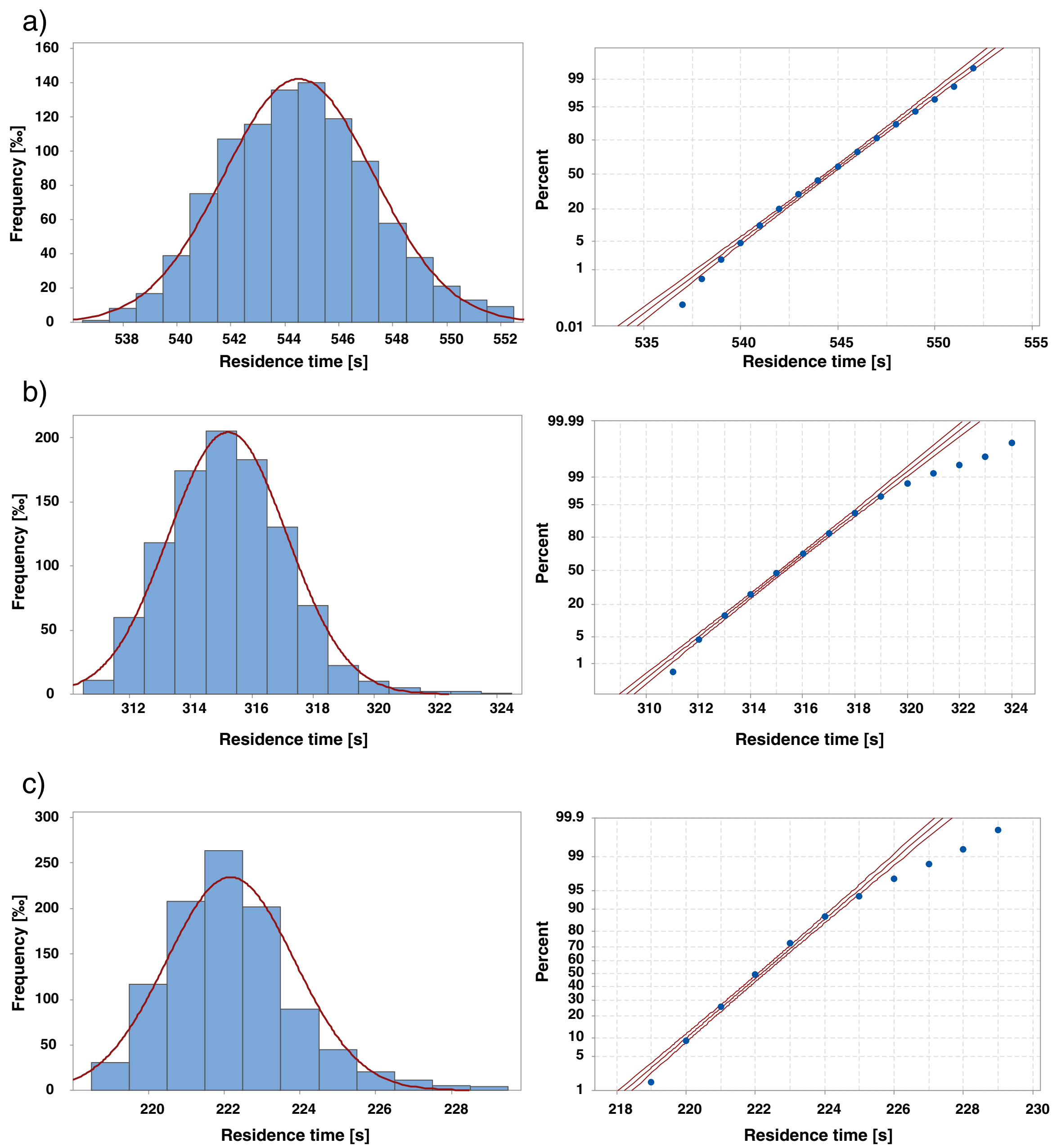

Fig. 6. Typical measured residence time distributions of flour at a motor speed of 740 rpm in 1 s intervals and overlaid normal distributions. Probability plots with 95% confidence intervals. a) Motor angle: 20°. b) Motor angle 30°. c) Motor angle: 40°.

rather than as being fully representative of the range of data possible at the particular condition.

In conclusion, a method was established to accurately measure the residence time distributions of flour in the vibrating machine. The RTDs can be approximated by normal distributions. The residence time decreases considerably with increasing motor angle and the distribution is narrower and thus more precise for 20° and 30° in comparison to 40° with respect to the mean.

3.2. Residence time development with time

The residence time of flour was tested for motor angles of 20°, 30°, and 40° and motor speeds of 600 rpm and 740 rpm over a time period of ca. 3.5 h. Each experiment took approximately 1 h. A marker sample was introduced to the product flow (100 kg/h) in intervals of 10 min and the shortest residence time was measured. Generally, between 3 and 4 experiments were performed per day and after each experiment, the flour was recirculated. Hence, the machine run-time is not continuous, but the cumulative run-length from the individual experiments per day is known. Before each day's experiments, the pipe was cleaned with a cleaning pig and water.

Fig. 8 shows that at motor speed of 740 rpm, the residence time increases over the period of 3.5 h for all motor angles. The data can be approximated with first order polynomials and the gradients increase from 0.09 s/min and 0.08 s/min at 40° and 30° to 0.3 s/min at 20°. The initial level of the residence time increases from 200 s to 260 s to 445 s for motor angles of 40°, 30°, and 20°, respectively. Within 3.5 h, the residence time increases by 62 s, 17 s and 15 s (calculated from the 2 samples over 210 min) for motor angles of 20°, 30°, and 40°, an increase of 13.9%, 6.5%, and 7.7% of the initial value, respectively. Thus, changing the motor angles from 20° to 30° has a bigger effect on initial level and slope of the residence time than varying them from 30° to 40°.

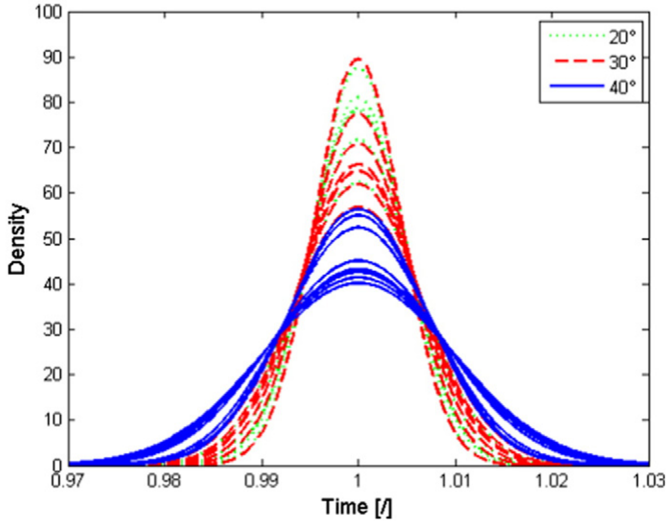

Fig. 7. Approximated normal distributions of the residence time of flour at motor angles of 20°, 30°, and 40° at 740 rpm.

At a motor speed of 600 rpm, results are more complex. At motor angles of 20°, the flour did not move up the spiral, but overflowed at the inlet. This demonstrates that as the acceleration at low motor angles is mainly vertical, the horizontal component of the acceleration can be insufficient to transport the flour. Fig. 9a presents the residence time for motor angles of 30° and 40° at 600 rpm. In contrast to 740 rpm, the residence time does not increase constantly over time. At 30°, the lowest residence time decreases by 12 s over the timescale of the experiment whereas at 40°, the residence time decreases slightly at first before it increases again in the end changing by 4 s. In both cases, the data can be described accurately by second order polynomials. This suggests different residence time dynamics at 600 rpm from those at 740 rpm. However, as at 740 rpm, the initial residence time decreases with increasing motor angle from 315 s at 30° to 250 s at 40°.

When experiments at motor angles of 40° were continued for another 3.5 h on a second day (without cleaning the pipe), the residence time maintained its increase (cf. Fig. 9b). The residence time on the second days at 600 rpm shows an increasing trend, approaching the results at 740 rpm.

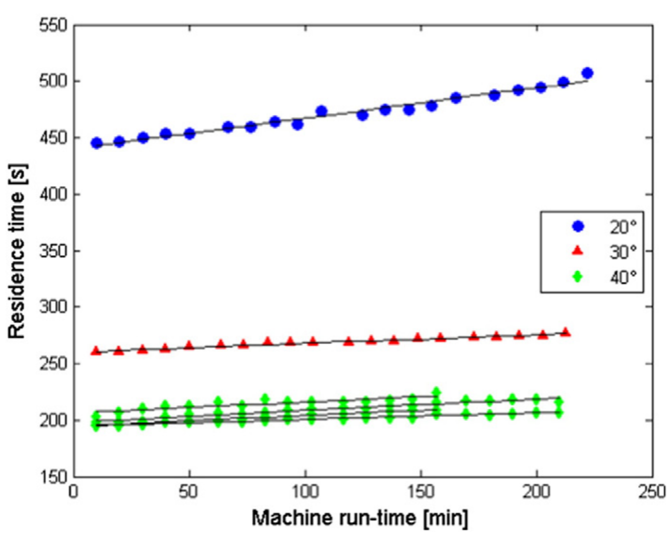

Fig. 8. Residence time of flour at motor angles of 20°, 30°, and 40° and a motor speed of 740 rpm with fitted 1st order polynomials.

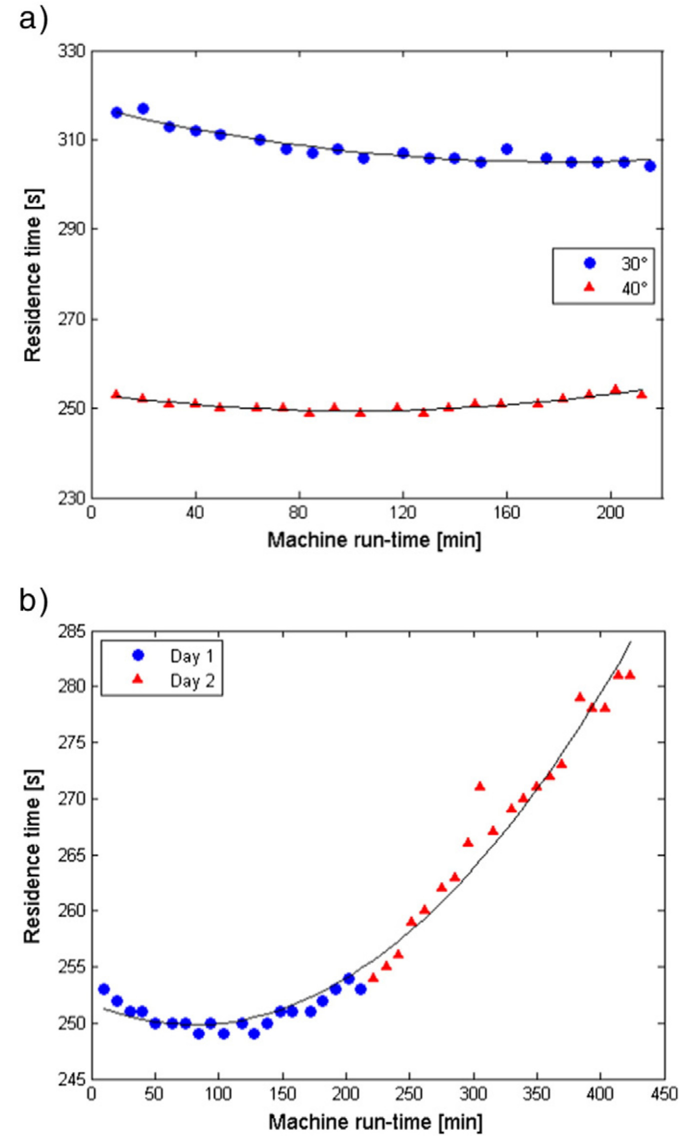

Fig. 9. a) Residence time of flour at motor angles of 30° and 40° and a motor speed of 600 rpm with fitted 2nd order polynomials. b) Motor angles of 40° and 600 rpm, showing (i) Day 1, starting with a clean pipe followed by (ii) Day 2, continuing without cleaning the pipe.

Fig. 10 depicts the influence of the motor speed on the residence time at a motor angle of 30°. Whilst the dynamics are different at 600 rpm and 740 rpm as explained above, the initial level of the residence time increases from 260 s at 740 rpm to 315 s at 600 rpm. The same was observed at 40° with residence times of 200 s and 253 s for 740 rpm and 600 rpm, respectively (data not shown). Hence, the residence time decreases with increasing motor angle and increasing motor speed. The dynamics are affected by the motor speed.

To demonstrate residence time dynamics, data from experiments run over 18 experimental days under the same conditions of 40° and 740 rpm is shown in Fig. 11. In this case both clean and used pipes were used. In all cases the residence time increased over time, in addition Fig. 11 shows that the initial residence time varies between 192 s and 228 s. The average increase over 3.5 h of machine run-time was 13.8 ± 5.0 s, equivalent to 6.6 ± 2.2% of the initial value (calculated from the days with 20 samples). The spread of residence time (56 s) was between 192 s and 248 s, which represents 29% of the minimum and 23% of the maximum

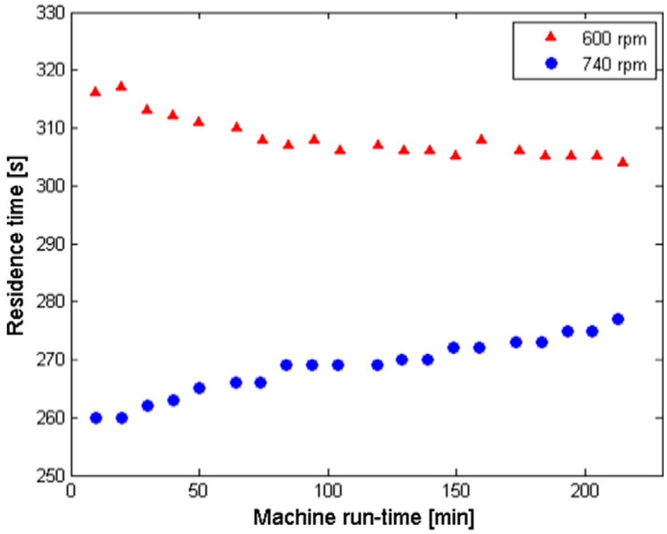

Fig. 10. Residence time of flour at a motor angle of 30° and motor speeds of 600 rpm and 740 rpm.

residence time. This spread is much larger than that observed in the individual distributions (i.e. standard deviation 1.88 s at 40°). It is necessary to identify the causes of this effect to accurately control and correctly operate the device.

From this data, three major questions arise:

i) What are the influences on the initial level of the residence time?
ii) What is responsible for the extent of the increase in residence time?
iii) Is there a plateau, where the residence time levels off?

Preliminary experiments showed no correlation between the residence time and ambient temperature, relative and specific humidity. Potential influences related to the product and the machine are discussed below.

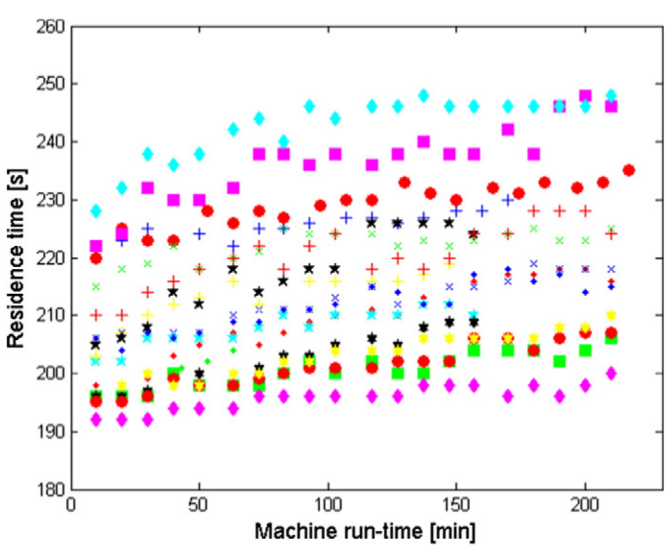

Fig. 11. Results from 18 different experiments for the residence time of flour over time at motor angles of 40° and a motor speed of 740 rpm. Different markers relate to different experimental days.

4. Investigation of variation in residence time

4.1. Influence of particle size

Separation of the product in the hopper or the vibrating tube could affect the residence time during an experiment. As the flour is recirculated after each experiment, any change of particle size distribution throughout the day could affect the residence time, for example by deposition of fine particles in the pipe or their loss at the outlet of the pipe, where the product is collected.

Table 1 presents the 10th, 50th, and 90th percentile of the particle size distribution of (i) the first flour sample of the day after 10 min of machine run-time and (ii) the last sample after 160 min. The *p*-values of an ANOVA between the samples are 0.03, 0.1, and 0.8 for x_{10}, x_{50}, and x_{90}, respectively. The results indicate no significant differences in particle size and that the particle size distribution remains constant throughout the day. Hence, product separation does not occur and does not cause the increase in residence time.

The flour used was sifted to a fine fraction (largely <75 μm) and a coarse fraction (largely >75 μm) (cf. Fig. 12a) to assess the effect of different sized fractions on the residence time. The results are shown in Fig. 12b, which also shows the scattered data of Fig. 11. The fine fraction was more cohesive than the coarse fraction, whereas the coarse fraction was particularly dusty and free flowing. This might explain why the average residence time is greater for the fine fraction (191 s) compared to the coarse fraction (187 s).

Both fractions however exhibit residence times below all data points measured for flour, apart from at the beginning and the end of the traces. The findings suggest it is not a particular size fraction that causes the observed phenomenon. Yet, the results suggest complex interactions between particles of different sizes as the residence times of both size fractions are dissimilar to that of flour.

4.2. Influence of water content of the product

Changes in water content of the flour could affect cohesion properties or the coefficient of restitution between the flour and the vibrating tube. The moisture content was determined of 15 samples within 3 experiments of 1 day. The average moisture content of all samples was 12.12 ± 0.03% (wet basis) and the data was randomly distributed. These findings prove that the water content of the product is not responsible for the increase in residence time.

4.3. Influence of static electricity

Charge effects are well known to cause problems in the flow of particles (Chubb, 2010). Interactions between the flour and the steel pipe may create static electricity. The flour might become charged over time. Alternatively, a thin layer of flour might deposit inside the pipe and become charged. The metal pipe cannot get charged as it has an electrical resistance of only 2.8 MΩ, which is easily overcome by the charge, and no change in the residence time was detected when the pipe was earthed.

Measurements of flour samples from the inlet of the pipe showed that the charge is close to zero (27 ± 23 pC/g) throughout the day

Table 1
Particle size distributions of flour with time.

Machine run-time [min]	Particle size		
	x_{10} [μm]	x_{50} [μm]	x_{90} [μm]
10	32.70 ± 0.23	86.98 ± 0.76	171.53 ± 0.90
160	33.22 ± 0.13	88.00 ± 0.32	171.69 ± 0.67

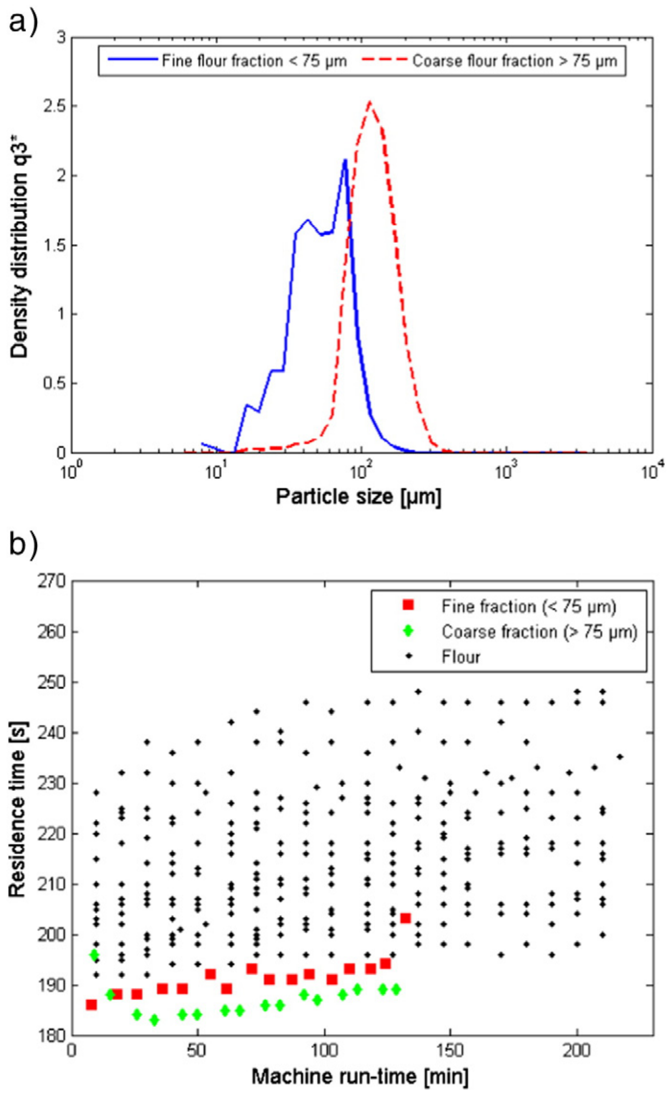

Fig. 12. a) Particle size distribution of fine and coarse flour fractions after sieving (hole size 75 µm). b) Residence time of flour, the fine flour fraction (<75 µm), and the coarse flour fraction (>75 µm) at motor angles of 40° and a motor speed of 740 rpm. Small points are the residence time data for flour shown in Fig. 11.

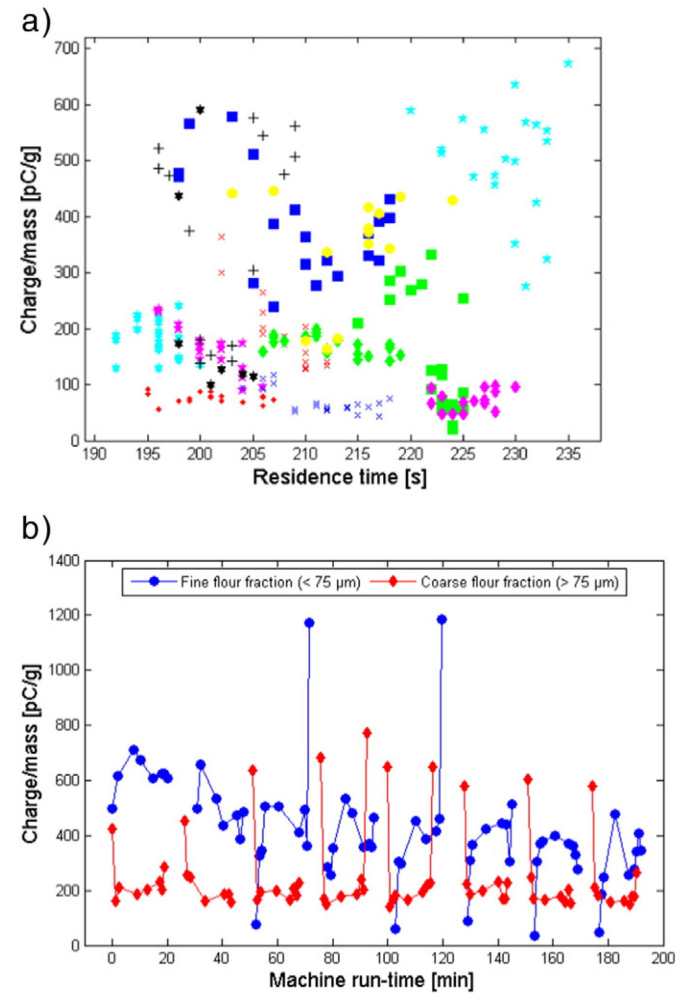

Fig. 13. a) Charge of flour samples from the outlet of the flexible pipe in correlation with the measured residence time. Different markers relate to different experimental days. b) Charge development of fine and coarse flour fractions over time. Markers of samples within one experiment are connected.

(data not shown). In contrast, charge measurements of flour at the outlet of the spiral were 220 ± 160 pC/g (cf. Fig. 13a). This proves that static charging of the material takes place in the pipe. However, no correlation could be established between the residence time and the outlet charge (Spearman's rho = 0.033). The findings suggest that the build-up of static charge in the bulk material does not cause the increase in residence time.

Fig. 13b illustrates the development of the outlet charge during the day for the fine flour fraction (<75 µm) and the coarse flour fraction (>75 µm) (cf. Section 4.1). The results indicate that the fine fraction is generally higher charged than the coarse fraction, which can be expected due to a larger surface area where charge may be separated. It is worth noting that during each experiment, the charge progresses in u-shaped curves for the coarse fraction. This behaviour is roughly mirrored by the fine fraction. The first and the last particles of each experiment have more contact with the wall than the flow in between, which could play a role in this context. Furthermore, the overall charge is rather constant during the day for the coarse fraction, whereas it decreases for the fine fraction. The reasons for this phenomenon are not clear.

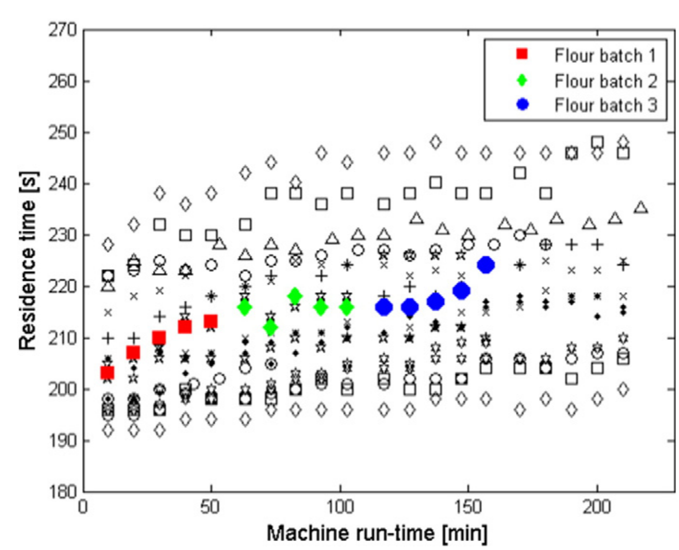

Fig. 14. Residence time of 3 fresh flour batches in comparison to recirculated flour at motor angles of 40° and a motor speed of 740 rpm. Different markers relate to different experimental days.

4.4. Influence of recirculation of the product

In the experiments, the product is recirculated for each experiment (1 h). This could cause errors in the results if the product is changed by the process in any way (e.g. particle size, electrostatic charge, etc.).

Fig. 14 shows the residence time of 3 fresh batches of flour together with the experiments of Fig. 11 where only one batch of flour was recirculated. The initial level of the residence time of the first batch was measured at 203 s, which is amongst the control data (192 s–228 s). Furthermore, the slope of a first order polynomial through the curve described by the 3 flour batches is 0.1 s/min. This is similar to the average gradient of the control data of 0.07 $\pm$ 0.03 s/min.

This in combination with the findings about particle size, water content, and charge suggests that alterations of the material do not cause the shift in residence time.

4.5. Influence of the cleanliness of the pipe

Deposition of product in the pipe might either obstruct the product flow or become electrostatically charged. To investigate this matter, 3 different cleaning methods were applied to the pipe and the residence time of flour was measured:

i) Cleaning with a cleaning pig and compressed air,
ii) Cleaning with a cleaning pig and water,
iii) Cleaning with a cleaning pig, water, and hand dishwashing liquid.

In the following, a dirty pipe is defined as one that has been used for flour and has not been cleaned before new experiments started.

First, the pipe was cleaned with a cleaning pig and compressed air after each 1 h experiment (3 times per day). This did not affect either the initial level of the residence time or the extent of the increase during the day (data not shown). Hence, flour accumulations as physical obstructions can be excluded to be the cause for the shift in residence time.

Second, Fig. 15 presents the residence times of flour for the pipe cleaned with a cleaning pig and water compared to a dirty pipe. Using a clean pipe reduces the initial level of the residence time, but to various extents as the starting points are not identical. Additionally, the gradient of the increase in residence time was determined to be 0.09 $\pm$ 0.02 s/min for the clean pipe (4 data sets) and 0.07 $\pm$ 0.03 s/min for the dirty pipe (14 data sets). Analysis of variance indicates no differences between both conditions ($p = 0.29$).

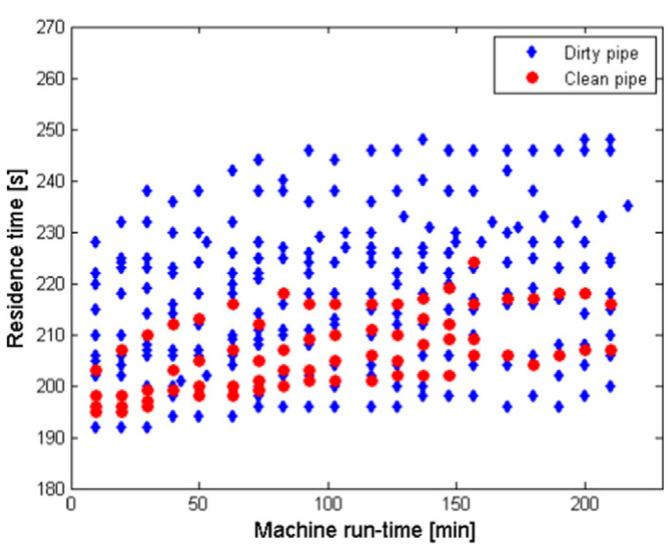

Fig. 15. Residence time of flour at motor angles of 40° and a motor speed of 740 rpm using a clean and a dirty pipe.

Cleaning with water has a more significant effect on the initial residence time than cleaning with air.

A line or deposit axially along the pipe could be seen in the pipe after flour had passed through. This supports the theory of the build-up of a thin, but visible flour layer. This line is not observable after cleaning. This does not mean that there cannot be a few particles adhering to the pipe wall. It may be that minor deposits of particles resulting from differences in cleaning effect might account for dissimilar initial levels of the residence time.

Third, judging from these outcomes, the pipe was cleaned with a hand dishwashing liquid, water, and a cleaning pig to ensure that all particles were removed. The results were similar to those obtained by cleaning with water only (data not shown). The slope of the increase in residence time was equivalent as well. This suggests either the adhesion of fine particles in the pipe is not the problem or the chosen method is inappropriate to remove the remaining particles.

4.6. Machine performance

The motors or other parts of the machine might warm up over time, which can potentially affect the vibrational frequency or deflection. This in turn could impact on the development of the residence time over the course of the day.

Tests were carried out in which the machine was run without product for 2 h before product flow was started. Results were compared to those where the machine was run with product from the beginning. The underlying hypothesis is that if the warming up of the machine is responsible for the increase in residence time, the latter would be expected to start at a higher initial residence time.

The results are shown in Fig. 16, and show that the residence time started at a lower value after the machine ran idle for 2 h in comparison to the control day. This suggests that the warming up of the machine does not cause the residence time to increase.

4.7. Extended run-length experiments

Fig. 17 shows the development of the residence time of flour over 4 consecutive experimental days and a further day of measurements after a weekend. It is evident that the residence time increases continuously over the first 4 days. This can be approximated with a second order polynomial ($R^2 = 0.97$). The slope decreases over the last 2 of the 4

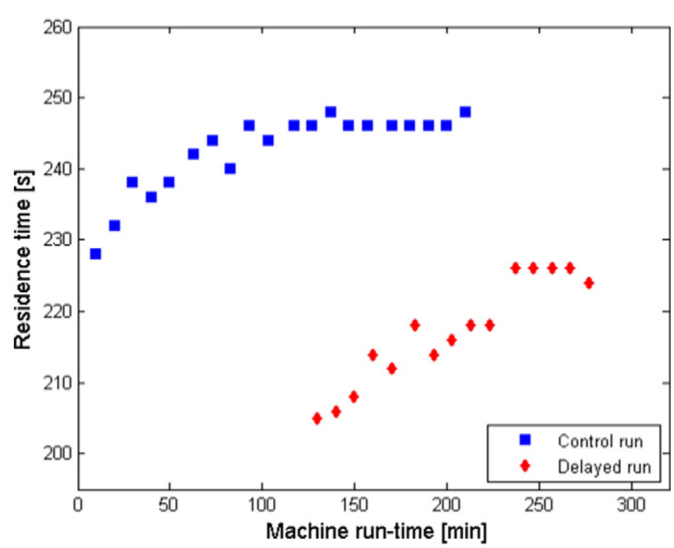

Fig. 16. Residence time of flour at motor angles of 40° and a motor speed of 740 rpm. (i) Control run where machine and product flow were started at the same time and (ii) delayed run where the machine was run for 2 h without product before the product flow was started.

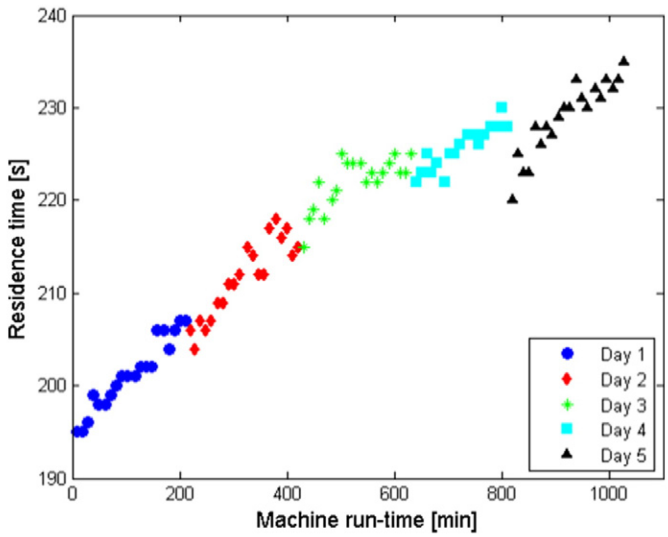

Fig. 17. Residence time of flour at motor angles of 40° and a motor speed of 740 rpm over a time period of 5 experimental days.

consecutive days. After the 2-day break, the residence time drops visibly and the subsequent increase over the course of the day is comparable with the first day of the experimental data set.

No constant value of the residence time was found. If the residence time increase is due to built-up of a thin flour layer inside the pipe, it would be expected to reach a saturation thickness at which the residence time levels off. Thus, the phenomenon appears more complex than a simple correlation between the residence time and the thickness of the layer on the inside of the pipe.

5. Conclusions

A novel tubular apparatus for the continuous heat treatment of flour has been studied. The residence time distributions of flour were characterised for various processing conditions and the development of the residence time in extended operation was analysed.

A method was established to accurately determine the residence time distributions. The RTDs can be approximated by normal distributions. The residence time decreases considerably with increasing motor angle and the distribution is narrower and thus more precise for 20° and 30° in comparison to 40° with respect to the mean. The width of the distribution is a critical factor for the accuracy of a thermal process.

The dynamics of the residence time were greatly affected by the motor speed. The systematically increasing residence time at 740 rpm could be fitted by a first order polynomial, whereas at 600 rpm, the residence time decreased at first, before an increase was observed and a second order polynomial was approximated.

The residence time increases over the course of approximately 3.5 h of machine run-time by 13.9%, 6.5%, and 7.7% for motor angles of 20°, 30°, and 40° and a motor speed of 740 rpm at room temperature. Several hypotheses concerning the product, the machine, and the environment were tested regarding the cause for this phenomenon.

Changes in the bulk material passing through the spiral were not the cause of the increase. This was established by the fact that the same trend was observed whether 3 fresh batches of product were used or the same product was recirculated during the day. Furthermore, particle size distributions and water contents were constant throughout the day. Electrostatic charging of the flour does take place during the process, but no correlation between charge of the material and residence time could be found. It is likely that electrostatic charging is relevant to the average time particles spend in transit.

The mechanical performance of the machine is constant over time and no correlation between residence time and ambient temperature and humidity could be found. It is sensible to assume the reason for the increase in residence time is in the pipe. It was found that two things led to a reduction in initial residence time: i) cleaning the pipe with a cleaning pig and water and ii) time, during which the machine is not running.

Cleaning the pipe with the pig and water reduces the initial residence time in contrast to cleaning with the pig and air. This suggests that it must be a very thin layer or few particles that stick firmly to the walls of the pipe that cause the residence time to shift. Overall, whichever process causes the residence time to increase during the day is reversible by cleaning the pipe or by idle time.

These observations suggest that a layer of few particles on the inside of the pipe wall causes the residence time to change. That the effect decreases when the equipment is left idle suggests an effect of something like electrostatics which discharges slowly over time.

More work is needed to identify the cause for the drift in residence time and to predict the residence time over periods of extended operation. The data does suggest that cleaning reduces the overall effect, and that frequent cleaning can allow relatively uniform behaviour. In practise, tests need to be done to confirm the residence time and ensure process uniformity.

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